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Karbala International Journal of Modern Science 2 (2016) 56–62

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Synthesis, assessment of substituent effect and antimicrobial activities of (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds

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Received 19 December 2015; revised 19 January 2016; accepted 19 January 2016

Available online 19 February 2016

Abstract

A series of substituted (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds has been synthesized from 4-aminoantipyrine and substituted benzaldehydes. Their structures have been confirmed by their physical constants, UV, IR and NMR spectral data. The observed UV absorption maximum λ_{max}(nm), IR frequencies νC═N(cm^{-1}), NMR δ(ppm) of C−H & C═N chemical shift values have been correlated with Hammett substituent constants and *F* and *R* parameters using single and multi-linear regression analyses. From the results of statistical analysis, the effect of substituents on the spectral data has been studied. The antimicrobial activities of all the Schiff bases synthesized have been studied using Bauer−Kirby method.

Keywords: Synthesis; UV, IR and NMR spectra; Correlation analysis; Antimicrobial activities

1. Introduction

Schiff's base compounds have been derived using condensation reaction of aldehydes or ketones with primary amino compounds. They contain −N═CHR group. Many Schiff's bases have been synthesized from heterocyclic compounds [1]. Schiff's bases and their metal complexes have been reported to exhibit vast applications in biological systems [2,3].

Though Schiff's bases contain a heterocyclic nucleus have efficient biological activities, the study of spectral correlation of these compounds has not been done well so for [4–6]. Antipyrine is used in the field of medicine [7] very much and it is believed that its amino derivative would equally be of important in drug industries possibly as intermediates in antipyretic and analgesic drugs [8].

In recent years, correlation analysis has been applied by chemists [9–12] for assessing the effect of substituents of Schiff's bases through spectral data.

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Peer review under responsibility of University of Kerbala.

http://dx.doi.org/10.1016/j.kijoms.2016.01.004

Literature survey shows that there is a little information available regarding the study of UV, IR and NMR spectral correlation and antimicrobial activities of substituted (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds. Hence the authors have taken efforts for synthesizing (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds and studying the effect of substituents through the spectral data as well as their antimicrobial activities.

2. Experimental

2.1. General

In the present investigation, all the chemicals used for synthesis have been procured from Sigma–Aldrich and E-Merck chemical companies. Mettler-FP51 melting point apparatus has been used for the observation of melting points of all (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds in open glass capillaries and are uncorrected. The SHIMADZU-1650 SPECTROMETER instrument has been utilized for recording UV spectra of all synthesized compounds using spectral grade methanol solvent. For recording infrared spectra (KBr, 4000–400 cm^{-1}) of these compounds AVATAR-300 Fourier transform spectrophotometer has been used. The NMR spectra of all the synthesized compounds have been recorded using BRUKER 400 spectrometer operating at 500 MHz for ^{1}H NMR spectra and 125.46 MHz for ^{13}C NMR spectra in $CDCl_3$ solvent using TMS as internal standard.

2.2. Synthesis of substituted (4E)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-ones

Equimolar quantities of 4-amino-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one (0.01 mol) and substituted benzaldehydes (0.01 mol) were stirred well with 0.5 ml of acetic acid and the mixture refluxed [13] for 10–12 h at 70–80 °C. The completion of the reaction was monitored by TLC continuously. Then the reaction mixture was cooled to room temperature and poured into crushed ice with constant stirring. Nearly yellow–orange precipitate settled down which was filtered and washed several times with cold water and recrystallized from ethanol to collect glittering pale yellow solid. The general reaction is as shown in Scheme 1.

3. Results and discussion

3.1. Correlation analysis of substituted (4E)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds

3.1.1. Uv–visible spectral correlations

The assigned UV absorption maximum λ_{max} (nm) values of all the substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds are presented in Table 1. These UV spectral values are correlated with Hammett substituent constants and *F* and *R* parameters using single and multilinear regression analyses [9–12,14]. Hammett equation employed for the correlation analysis, involving the UV absorption maximum is shown in equation (1).

$$\lambda = \rho \cdot \sigma + \lambda_0 \quad (1)$$

where λ_0 is the absorption maximum of the parent member of this series.

The results of statistical analysis [9–12,14] are shown in Table 2. From the Table it is evident that the UV absorption maximum λ_{max} (nm) values of all the substituted (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-ones except those with 3-Br and 3-Cl substituents have shown satisfactory correlations with Hammett constant σ (r = 0.900), σ^+ (r = 0.900) and σ_R (r = 0.904) and *R* (0.904) parameter. The remaining Hammett constants have shown poor correlations. This is due to the inability of substituents for predicting the reactivity on the absorption and it is associated with resonance-conjugative structure as shown in Fig. 1. However the multi-correlation analysis produce satisfactory correlations as shown in equations (2) and (3).

$$\lambda_{max}(\mathrm{nm}) = 255.050\,(\pm 22.89) + 30.539\,(\pm 9.88)\,\sigma_I - 74.801\,(\pm 16.84)\,\sigma R \quad (2)$$

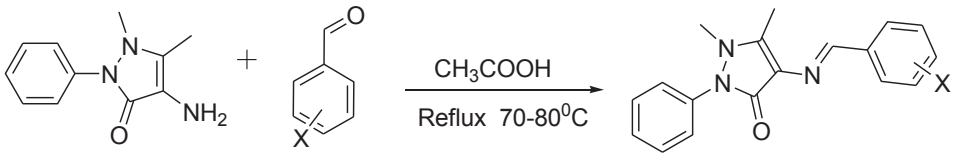
$(R = 0.948,\ n = 10,\ P > 95\%)$

$$\lambda_{max}(nm) = 259.702\,(\pm 22.94) + 17.667\,(\pm 6.463) \times F - 54.679\,(\pm 10.37)\,R \quad (3)$$

$(R = 0.948,\ n = 10,\ P > 95\%)$

3.1.2. IR spectral correlation

The assigned infrared stretching frequency ν C=N (cm^{-1}) values of all the substituted (4E)-4-(benzyli-



X=H, 3-Br,4-Br,3-Cl,4-Cl,4-F,4-CH_3,4-OCH_3,3-NO_2,4-NO_2

Scheme 1. Synthesis of substituted (4E)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds.

Table 1
The UV, IR and NMR spectral data of substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-ones (entries: 1–10).

Entry	X	M.F.	M. W.	M.p. (°C)	UV λ_{max} (nm)	IR νC=N (cm^{-1})	NMR δ^1H CH=N (ppm)	NMR δ^{13}C C=N (ppm)
1	H	$C_{17}H_{17}N_3O$	279	159–160 (160) [15]	249.00	1570.06	9.761	160.86
2	3-Br	$C_{17}H_{16}BrN_3O$	358		335.50	1564.27	9.682	160.59
3	4-Br	$C_{17}H_{16}BrN_3O$	358	169–170 (168) [16]	263.00	1564.27	9.701	160.74
4	3-Cl	$C_{17}H_{16}ClN_3O$	313	188–189	332.50	1562.34	9.700	160.65
5	4-Cl	$C_{17}H_{16}ClN_3O$	313	253–254 (255) [17]	259.50	1566.20	9.717	160.73
6	4-F	$C_{17}H_{16}FN_3O$	297	119–120 (120) [18]	290.00	1597.06	9.710	162.82
7	4-CH_3	$C_{18}H_{19}N_3O$	293	136–137	260.50	1581.63	9.695	161.46
8	4-OCH_3	$C_{18}H_{19}N_3O_2$	309	151–152	281.00	1589.34	9.729	160.95
9	3-NO_2	$C_{17}H_{16}N_4O_3$	324	218–219 (218) [29]	235.00	1583.56	9.811	160.46
10	4-NO_2	$C_{17}H_{16}N_4O_3$	324	252–253 (254) [19]	266.50	1562.34	9.802	160.27

deneamino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds are presented in Table 1. These infrared stretching frequency values are correlated with different Hammett substituent constants and *F* and *R* parameters using single and multi-linear regression analyses.

The Hammett equation employed for the structure parameter correlation involving group frequencies, is shown in equation (4).

$$\nu = \rho\sigma + \nu_0 \tag{4}$$

where ν_0 is the frequency of the parent member of this series.

The results of the statistical analysis [9–12,14] are presented in Table 2. From this table it is evident that the infrared stretching frequency νC=N (cm^{-1}) values of all the substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds except those with 4-F, 3-NO_2 and 4-NO_2 substituents have shown satisfactory correlations with Hammett constants σ (r = 0.904) and σ^+ (r = 0.905). The remaining Hammett constants have shown poor correlations. But the multi-correlation analysis reveals satisfactory correlations as shown in equations (5) and (6).

$$\begin{aligned}\nu C = N\left(cm^{-1}\right) = {} & 1571.205(\pm 8.528) \\ & - 3.362(\pm 0.938)\sigma_I \\ & - 30.829(\pm 3.060)\sigma_R\end{aligned} \tag{5}$$

$$(R = 0.951,\ n = 10,\ P > 95\%)$$

$$\begin{aligned}\nu C = N\left(cm^{-1}\right) = {} & 1567.025\,(\pm 7.935) \\ & + 3.920\,(\pm 1.720)\,F \\ & - 28.854\,(\pm 5.034)\,R\end{aligned} \tag{6}$$

$$(R = 0.956,\ n = 10,\ P > 95\%)$$

3.1.3. NMR spectral correlation

The observed chemical shift values (ppm) of all the substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-

Table 2
Results of statistical analysis of UV λ_{max} (nm), ν C=N (cm^{-1}) IR, NMR δ^1H (ppm) CH=N and δ^{13}C (ppm) C= N of substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-ones.

Frequency	Const.	r	I	ρ	s	n	Correlated derivatives
$\lambda_{max(nm)}$	σ	0.900	277.48	−0.768	35.51	8	H, 4-Br, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ^+	0.900	277.31	−0.037	35.51	8	H, 4-Br, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ_I	0.801	268.54	22.292	35.04	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ_R	0.904	267.69	−70.164	31.99	8	H, 4-Br, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	F	0.824	269.25	19.740	35.12	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	R	0.904	266.78	−55.376	32.42	8	H, 4-Br, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
νC=N (cm^{-1})	σ	0.904	1578.06	−16.890	11.92	7	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-CH_3, 4-OCH_3
	σ^+	0.905	1576.67	−15.238	11.01	7	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-CH_3, 4-OCH_3
	σ_I	0.866	1576.76	−6.761	13.36	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ_R	0.843	1569.81	−31.340	11.59	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	F	0.884	1572.06	−5.013	13.41	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	R	0.872	1568.60	−29.009	11.14	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
δCH=N (ppm)	σ	0.942	9.685	−0.104	0.08	7	3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3,4-OCH_3,
	σ^+	0.935	9.699	−0.063	0.08	7	3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3,4-OCH_3,
	σ_I	0.900	9.709	−0.0003	0.09	7	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-OCH_3
	σ_R	0.908	9.760	0.335	0.05	8	H, 3-Br, 4-Br, 3-Cl, 4-Cl,4 −CH_3, 3-NO_2, 4-NO_2
	F	0.863	9.740	−0.073	0.08	10	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-F, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	R	0.907	9.760	0.276	0.05	8	H, 3-Br, 4-Br, 3-Cl, 4-Cl,4 −CH_3, 3-NO_2, 4-NO_2
δC=N (ppm)	σ	0.980	161.02	−0.887	0.24	9	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ^+	0.957	161.18	−0.924	0.33	9	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ_I	0.957	161.17	−0.924	0.33	9	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-CH_3, 4-OCH_3, 3-NO_2, 4-NO_2
	σ_R	0.965	160.65	−1.233	0.30	8	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-OCH_3, 3-NO_2, 4-NO_2
	F	0.966	160.62	−1.048	0.30	8	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-OCH_3, 3-NO_2, 4-NO_2
	R	0.966	160.62	−1.048	0.30	8	H, 3-Br, 4-Br, 3-Cl, 4-Cl, 4-OCH_3, 3-NO_2, 4-NO_2

r = correlation coefficient; ρ = slope; I = intercept; s = standard deviation; n = number of substituents.

2,3-dimethyl-1-phenyl pyrazol-5-one compounds are presented in Table 1. These chemical shift values (ppm) are correlated with different Hammett substituent constants and *F* and *R* parameters using single and multi-linear regression analyses [9–12,14]. In this case the Hammett equation employed for the structure-parameter correlation is shown in equation (7).

$$\delta = \rho\sigma + \delta_0 \quad (7)$$

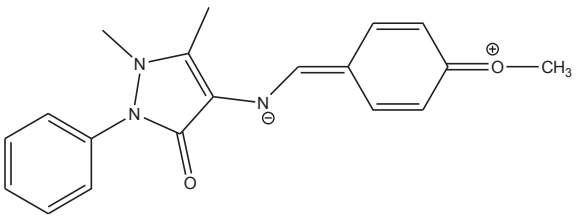


Fig. 1. Resonance-conjugative structure.

where δ_0 is the chemical shift of the corresponding parent compound.

3.1.3.1. ^1H NMR spectral correlation. From Table 2, it is evident that the ^{1}H NMR chemical shift δCH=N (ppm) values of all the (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one compounds except those with H, 3-NO_2 and 4-NO_2 substituents have shown satisfactory correlations with Hammett constants σ (0.904) and σ^+ (0.935). The Hammett constant σ_I has also shown satisfactory correlation (r = 0.900) for all the compounds except those with 4-CH_3, 3-NO_2 and 4-NO_2 substituents. All the compounds except 4-F and 4-OCH_3 have shown satisfactory correlations with Hammett constant σ_R (r = 0.908) and *R* (r = 0.907) parameter. The remaining *F* parameter has shown poor correlation (r < 0.900) with all the substituents. However the multi-correlation analysis reveals satisfactory correlations as shown in equations (8) and (9).

$$\delta\,(CH=N)\,(ppm) = 9.771\,(\pm 0.039) - 0.037\,(\pm 0.018)\sigma_I + 0.340\,(\pm 0.019)\,\sigma_R \quad (8)$$

$$(R = 0.981,\ n = 10,\ P > 95\%)$$

$$\delta\,(CH=N)\,(ppm) = 9.787\,(\pm 0.038) - 0.062\,(\pm 0.013)\,F + 0.274\,(\pm 0.077)\,R \quad (9)$$

$$(R = 0.981,\ n = 10,\ P > 95\%)$$

3.1.3.2. ^{13}C NMR spectral correlation. The results of the statistical analysis [9–12,14] are presented in Table 2. It is evident that the ^{13}C NMR chemical shift δC=N (ppm) values of all the (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds except that with 4-F substituent have shown satisfactory correlations with Hammett constants σ (r = 0.980) σ^+ (r = 0.957) and σ_I (r = 0.957). All the substituents except those with 4-F and 4-CH_3 substituents have also shown satisfactory correlations with the remaining Hammett constant σ_R (r = 0.965) and F (r = 0.966) and R (r = 0.966) parameters. The remaining *F* parameter has shown poor correlation (r < 0.900) with all the substituents. In each case, the reason for poor correlation is due to the polar, inductive, resonance and field effect of the substituents unable to predict their electronic effects through resonance as per the conjugative structure as shown in Fig 1.

The multi-correlation analysis shows satisfactory correlations as shown in equations (10) and (11).

$$\delta C = N(ppm) = 160.978\,(\pm 0.171) - 0.801\,(\pm 0.242) \times \sigma_I - 1.111(\pm 0.402)\sigma_R \quad (10)$$

$$(R = 0.982,\ n = 10,\ P > 95\%)$$

$$\delta C = N(ppm) = 160.853\,(\pm 0.187) - 0.590\,(\pm 0.372) \times F - 1.07\,(\pm 0.379)\,R \quad (11)$$

$$(R = 0.976,\ n = 10,\ P > 95\%)$$

3.2. Antimicrobial activities

3.2.1. Antibacterial sensitivity assay

The antibacterial activities of all synthesized substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds have been studied against 3 g positive pathogenic strains *S. aureus, B. substilis and M. lutes* and 2 g negative strains *E. coli* and *P. aeruginosa*. The disc diffusion technique has been followed using the Kirby–Bauer method [20] and ciprofloxcin was used as the standard. The observed zone of inhibition values are given in Table 3.

Table 3
Zone of inhibition (mm) values of antibacterial activity of substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-ones.

S.No.	Substituents	Zone of inhibition (mm)				
		Gram positive bacteria			Gram negative bacteria	
		B. subtilis	*M. luteus*	*S. aureus*	*E. coli*	*P. aeruginosa*
1	H	10	12	17	8	11
2	3-Br	21	20	19	16	19
3	4-Br	15	15	10	14	10
4	3-Cl	19	18	17	14	13
5	4-Cl	26	23	18	19	25
6	4-F	11	12	13	13	10
7	4-CH_3	18	17	18	14	15
8	4-OCH_3	21	23	21	16	17
9	3-NO_2	17	20	11	16	22
10	4-NO_2	24	22	20	18	19
Standard	Ciprofloxacin	31	30	32	34	28
Control	DMSO	0	0	0	0	0

Table 4
Zone of inhibition (mm) values of antifungal activities of substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds.

Entry	Substituents	Zone of inhibition (mm)		
		A. niger	*M. species*	*T. viride*
1	H	10	9	12
2	3-Br	8	8	8
3	4-Br	8	6	8
4	3-Cl	11	0	10
5	4-Cl	9	9	13
6	4-F	6	6	0
7	4-CH_3	9	9	8
8	4-OCH_3	10	8	9
9	3-NO_2	9	11	8
10	4-NO_2	12	8	12
Standard	Micnazole	14	15	16
Control	DMSO	0	0	0

Zone of inhibition (mm) values of antibacterial activity of substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds reveals that all the compounds have shown moderate to good activity against all the five microorganisms evaluated in the present investigation. Schiff's bases with 4-Cl and 4-NO_2 substitutend have shown good activity against *P. aurogenosa*. The remaining compounds have shown moderate activity.

3.2.2. Antifungal sensitivity assay

The antifungal activities of all the synthesized substituted (4*E*)-4-(benzylidene amino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds have been studied against three fungal species namely, *A. niger, M. species* and *T. viride*. The disc diffusion technique has been followed using the Kirby–Bauer method [20] and Micnazole was used as the standard. The observed zone of inhibition values are given in Table 4. All the compounds have shown moderate to good activity against all the three fungal species evaluated in general. The parent compound, 3-Cl and 4-NO_2 substituted Schiff's bases have shown good activity against *A. niger* and *T. viride*. The 4-Cl substituted Schiff's base has shown good activity against *T. viride*. In the case of *M. species*, the 3-Cl substituted Schiff's base has shown poor activity.

4. Conclusion

Using condensation method, a series of substituted (4*E*)-4-(benzylideneamino)-1,2-dihydro-2,3-dimethyl-1-phenyl pyrazol-5-one compounds has been synthesized and their structures have been confirmed by their physical constants and spectral data. The spectral data of these compounds have been correlated with Hammett sigma constants and *F* and *R* parameters using single and multi-linear regression analyses. Most of the single linear regression analyses with UV, IR and NMR spectral data have shown satisfactory correlations for all the substituents. Schiff's bases with 4-Cl and 4-NO_2 substitutend have shown good activity against *P. aurogenosa*. The remaining compounds have shown moderate activity. The parent compound, 3-Cl and 4-NO_2 substituted Schiff's bases have shown good activity against *A. niger* and *T. viride*. The 4-Cl substituted Schiff's base has shown good activity against *T. viride*. In the case of *M. species*, the 3-Cl substituted Schiff's base has shown poor activity. Some of the compounds have shown good antibacterial and antifungal activities. Most of the compounds have been shown moderate activity.

Acknowledgement

The authors thank DST NMR Facility, Department of Chemistry, Annamalai University, Annamalainagar-608002, for recording NMR spectra of all compounds.

Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.kijoms.2016.01.004.

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